

# Electromagnetic properties of microwave sintered $x\text{TiO}_2 + (1 - x) \text{CoFe}_2\text{O}_4$ nanocomposites

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**Abstract** The nanocomposites of  $x\text{TiO}_2 + (1 - x) \text{CoFe}_2\text{O}_4$  (where  $0 \leq x \leq 1$ ) were prepared using microwave–hydrothermal method at 165 °C/45 min. The as-synthesized powders were characterized using X-ray diffraction (XRD), transmission electron microscope and Fourier transform infrared spectroscopy. The particle size was found to be  $\sim 18$ ,  $\sim 22$  and 24 nm for  $\text{TiO}_2$ ,  $\text{CoFe}_2\text{O}_4$ , 50 mol%  $\text{TiO}_2 + 50$  mol%  $\text{CoFe}_2\text{O}_4$  composite powder, respectively. The as-prepared powders were densified at 500 °C/30 min using microwave sintering method. The sintered composite samples were characterized using XRD and field emission scanning electron microscopy. The bulk densities of the present composites were increasing with an addition of  $\text{TiO}_2$ . The saturation magnetization of composites decreased with an increase of  $\text{TiO}_2$  content. The grain sizes of all the composite lies between 54 and 78 nm. The addition of  $\text{TiO}_2$  to ferrite increased  $\epsilon'$  and  $\epsilon''$  and the resonant frequency of all the sintered samples were found to be  $>1$  GHz. The value of  $\mu''$  found to increase with an increase of  $\text{TiO}_2$ .

**Keywords** Microwave sintering · Dielectric properties · Magnetic properties

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## Introduction

Much attention has been paid to microwave absorption materials due to their unique absorbing microwave energy and promising applications (Shin and Oh 1993; Martha 2000; Peng et al. 2005). Extensive study has been carried out to develop new microwave absorbing materials with a high magnetic and electric loss (Ruan et al. 2000; Babbar et al. 2000; Verma et al. 2003; Cho et al. 1996; Singh et al. 2000; Nakamura 2000). To our knowledge, ferrites might be a candidate as the microwave absorbing materials because of their high-specific resistance, remarkable flexibility in tailoring the magnetic properties and ease of preparation (Martha 2000). In the past decades, the spinel ferrites have been utilized as the most frequent absorbing materials in various forms (Peng et al. 2005). Cobalt ferrite ( $\text{CoFe}_2\text{O}_4$ ) is a common spinel ferrite material and has been widely used in microwave applications. Recently, it has been shown that magnetic nano-composites are useful as microwave absorbing materials due to their advantages in respect to light weight, low cost, design flexibility, and microwave properties over pure ferrites (Singh et al. 2000; Kim et al. 1996). On the other hand, titanium dioxide ( $\text{TiO}_2$ ) is an important inorganic semiconductor and has temperature and environmentally stable dielectric properties (Dervos et al. 2004).

In the present investigation, the nanocomposites of  $\text{TiO}_2$ – $\text{CoFe}_2\text{O}_4$  with different mol% of  $\text{TiO}_2$  were prepared using microwave–hydrothermal (M-H) method. The advantage of M-H method is given elsewhere (Komarneni et al. 1996). The as-prepared powders were characterized using X-ray diffraction (XRD), transmission electron microscope (TEM) and fourier transform infrared spectroscopy (FTIR). The  $\text{TiO}_2$ – $\text{CoFe}_2\text{O}_4$  composite materials were sintered at 500 °C/30 min using microwave sintering method, which helps them to meet the need for low

temperature co-fired ceramics technology. The frequency dependence of complex permittivity and permeability of the present composites were studied in the range of 1 MHz–1.8 GHz and the obtained results were discussed in this paper.

## Experimental method

In the present investigation,  $x\text{TiO}_2 + (1 - x)\text{CoFe}_2\text{O}_4$  composites ( $0 \leq x \leq 1$ ) were prepared using appropriate amounts of  $\text{TiO}_2$  and  $\text{CoFe}_2\text{O}_4$  solutions. The  $\text{CoFe}_2\text{O}_4$  was prepared using cobalt nitrate [ $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ] and ferric nitrate [ $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ] in stoichiometric ratio. The  $\text{TiO}_2$  was synthesized using titanium tetrachloride ( $\text{TiCl}_4$ ) solutions. These reagents were dissolved in 50 ml of de-ionized water. To this solution, sodium hydroxide ( $\text{NaOH}$ ) was added to maintain the pH of the solution  $\sim 12$ . Controlling of pH is the key factor to synthesize the nanopowder.

The  $x\text{TiO}_2 + (1 - x)\text{CoFe}_2\text{O}_4$  composite precipitation was transferred into double-walled digestion vessels that have an inner liner and cover made up of Teflon PFA and an outer high strength layer made up of Ultem polyetherimide and then treated using M-H method at  $165^\circ\text{C}/45$  min. The M-H treatment was performed using a microwave accelerated reaction system (MARS-5, CEM Corp., Mathews, NC, USA). This system uses 2.45 GHz microwave frequency and can be operated at 0–100 % full power ( $1,200 \pm 50$  W). The reaction vessel was connected to an optical probe to monitor and control the temperature during synthesis. The product was separated by centrifugation and then washed repeatedly with de-ionized water, followed by drying in an oven overnight at  $100^\circ\text{C}$ . Thus, the obtained powders were weighed and the percentage yields were calculated from the total expected based on the solution concentration and volume and the amount that was actually crystallized. A 94 % of the yield was obtained. A similar procedure was followed to obtain different mol% of  $\text{TiO}_2$  composites.

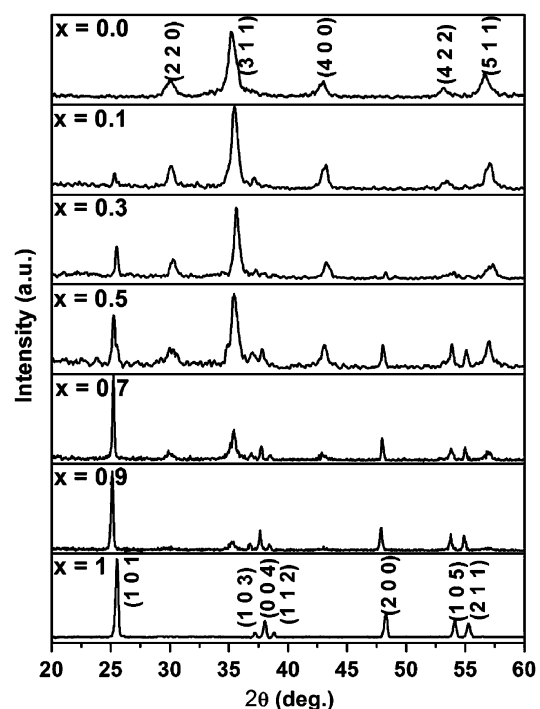
The phase identification of powders was performed using XRD with  $\text{Cu K}\alpha$  ( $\lambda_{\text{CuK}\alpha} = 1.54056 \text{ \AA}$ ) radiation. The particle size was calculated from TEM. The powders were uniaxially pressed into toroidal samples and pellets. The specimens were sintered at  $500^\circ\text{C}/30$  min using microwave sintering method (Murthy 2002). The microwave sintering process was carried out using a domestic microwave oven, operated at 2.45 GHz of frequency and an output power tunable up to 1,100 W. Temperature of the sample was measured using platinum sheathed Cr–Al thermocouple with an accuracy of  $\pm 1^\circ\text{C}$ . The temperature of the furnace was controlled with a PID controller. The sintering temperature was chosen for maximum ceramic

density without apparent chemical reaction as determined from XRD analysis.

The phase identification and grain distribution of the sintered samples were identified using XRD and field emission scanning electron microscope (FE-SEM). The bulk density of the present samples was measured using the Archimedes principle. The frequency dependent complex permittivity ( $\epsilon'$  and  $\epsilon''$ ) and permeability ( $\mu'$  and  $\mu''$ ) were measured in the range of 1 MHz–1.8 GHz using Agilent 4291B impedance analyzer at room temperature.

## Results and discussion

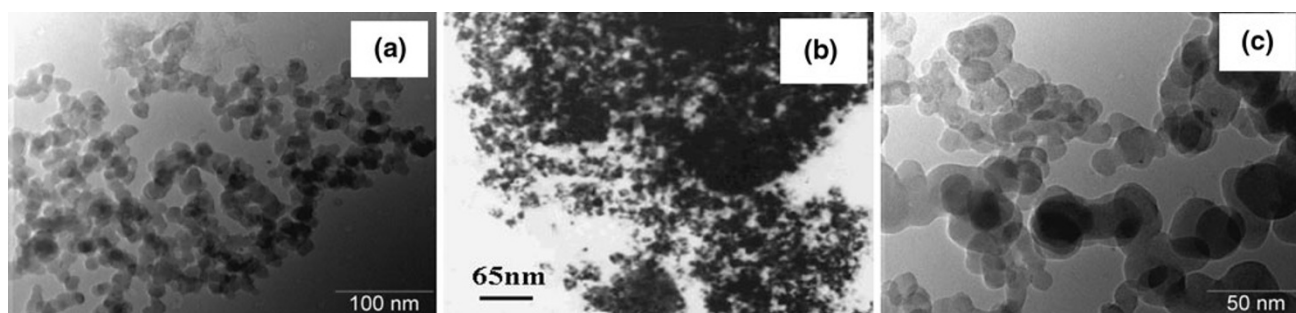
Figure 1 shows the XRD patterns of the as-synthesized powders of  $\text{TiO}_2$  ( $x = 1.0$ ),  $\text{CoFe}_2\text{O}_4$  ( $x = 0$ ) and  $x\text{TiO}_2 + (1 - x)\text{CoFe}_2\text{O}_4$  ( $x = 0.9\text{--}0.1$ ) composite powders, respectively. It can be seen from the figure that the powders possess anatase phase ( $x = 1$ ) (JCPDS card no. 89-4203) and spinel phase ( $x = 0$ ) (JCPDS card no. 03-0864), respectively. It can be seen from the figure that both  $\text{TiO}_2$  and ferrite peaks are present in the XRD patterns. No other phases were detected in the XRD patterns. The crystallite size ( $D_m$ ) of the  $\text{CoFe}_2\text{O}_4$  in the composite has been estimated with the help of (3 1 1) peak using Scherrer's equation:  $D_m = K\lambda/\beta\cos\theta$ , where  $K$  is a constant,  $\beta$  the full width half maxima,  $\lambda$  the wavelength of X-rays used and  $\theta$  is the diffraction angle. The crystallite



**Fig. 1** XRD patterns of microwave-hydrothermally synthesized  $x\text{TiO}_2 + (1 - x)\text{CoFe}_2\text{O}_4$  composites ( $0 \leq x \leq 1$ )

**Table 1** Data of lattice constant and crystallite size

Composition	Sample name	Lattice constant			Crystallite size of ferrite calculated from (3 1 1) peak (nm)
		Ferrite	TiO <sub>2</sub>		
		(a) (Å)	(a) (Å)	(c) (Å)	
TiO <sub>2</sub>	TO	–	3.701	9.499	–
0.9 mol% TiO <sub>2</sub> + 0.1 mol% CoFe <sub>2</sub> O <sub>4</sub>	CFT9	8.386	3.701	9.499	30
0.7 mol% TiO <sub>2</sub> + 0.3 mol% CoFe <sub>2</sub> O <sub>4</sub>	CFT7	8.382	3.701	9.502	26
0.5 mol% TiO <sub>2</sub> + 0.5 mol% CoFe <sub>2</sub> O <sub>4</sub>	CFT5	8.378	3.699	9.505	25
0.3 mol% TiO <sub>2</sub> + 0.7 mol% CoFe <sub>2</sub> O <sub>4</sub>	CFT3	8.372	3.699	9.505	23
0.1 mol% TiO <sub>2</sub> + 0.9 mol% CoFe <sub>2</sub> O <sub>4</sub>	CFT1	8.365	3.699	9.509	21
CoFe <sub>2</sub> O <sub>4</sub>	CF	8.361	–	–	20

**Fig. 2** TEM images of **a** TiO<sub>2</sub>, **b** CoFe<sub>2</sub>O<sub>4</sub> and **c** 50 wt% TiO<sub>2</sub> composite

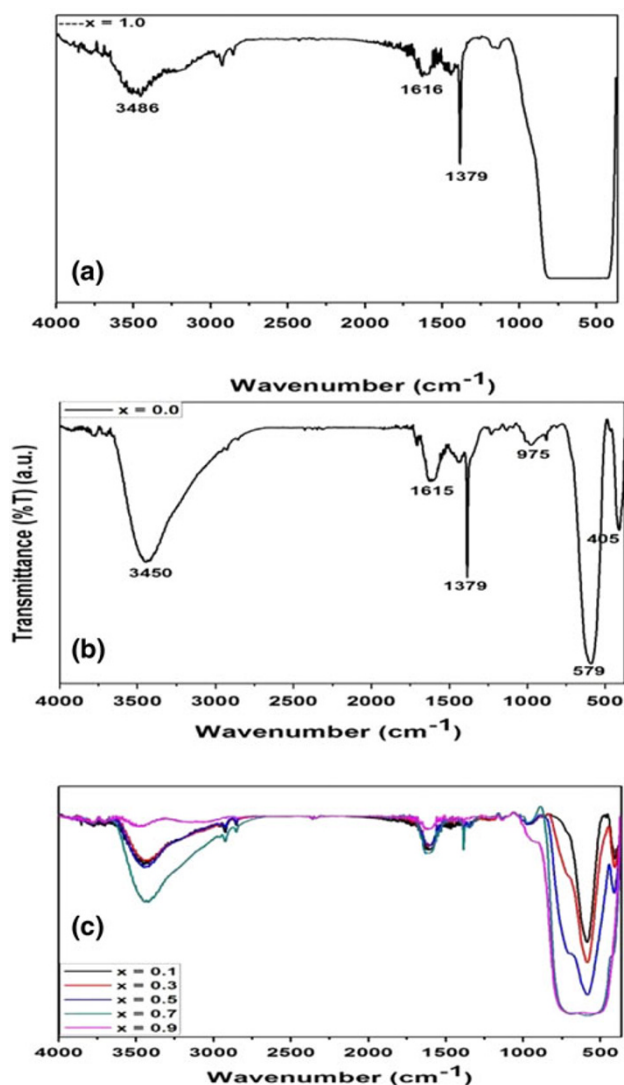
sizes of the ferrite in the composite and lattice constants of the composites were given in Table 1. It can be seen from the table that the crystallite size decreases and lattice parameters increase with an increase of TiO<sub>2</sub> content.

Figure 2a, b and c shows the TEM pictures of the as-synthesized TiO<sub>2</sub>, CoFe<sub>2</sub>O<sub>4</sub> and 0.5 mol% TiO<sub>2</sub> + 0.5 mol% CoFe<sub>2</sub>O<sub>4</sub> powders, respectively. From the TEM pictures, the average particle size was calculated and it was found to be ~18, 22 and ~24 nm for TiO<sub>2</sub>, CoFe<sub>2</sub>O<sub>4</sub> and 0.5 mol% TiO<sub>2</sub> + 0.5 mol% CoFe<sub>2</sub>O<sub>4</sub> powders, respectively.

Figure 3a, b and c shows the FTIR spectra of as-synthesized  $x$ TiO<sub>2</sub> + (1 –  $x$ ) CoFe<sub>2</sub>O<sub>4</sub> (0 ≤  $x$  ≤ 1) composite powders. Figure 3a shows the FTIR spectra of as-synthesized TiO<sub>2</sub> powders. The broad absorption in the 3,600–2,800 cm<sup>–1</sup> range, with a maximum at 3,486 cm<sup>–1</sup>, arises from the stretching vibrations of O–H group (i.e., involved in hydrogen bonds) and the symmetric and anti-symmetric  $\nu_{OH}$  modes of molecular water coordinated to Ti<sup>4+</sup> cations (Morterra 1988; Primet et al. 1971). The band at 1,616 cm<sup>–1</sup> is assigned to the molecular water bending mode. The band at 1,379 cm<sup>–1</sup> is ascribed to the anti-symmetric stretching vibration of NO<sub>3</sub><sup>–</sup>, arising from the residual nitrate. The broad peak in the range of 800–500 cm<sup>–1</sup> is identified to have the frequencies corresponding to bulk titania skeletal (Kumar et al. 2000).

Figure 3b shows the FTIR spectra of as-synthesized CoFe<sub>2</sub>O<sub>4</sub> powders. It can be seen from the figure that there are two main broad, in the range of 400–300 cm<sup>–1</sup> and 600–500 cm<sup>–1</sup>, bands are observed. The highest one,  $\nu_1$ , is generally observed in the range 600–500 cm<sup>–1</sup>, and it corresponds to intrinsic stretching vibrations of the metal at the tetrahedral site ( $T_d$ ),  $M_{tetra} \leftrightarrow O$ , whereas the  $\nu_2$ , lowest band is usually observed in the range 430–385 cm<sup>–1</sup>, is assigned to octahedral metal stretching ( $O_h$ ),  $M_{octa} \leftrightarrow O$  (Baykal et al. 2008; Patil et al. 1998). In the FTIR spectrum, the band at 579 cm<sup>–1</sup> is assigned as  $\nu_1$  ( $M_{tetra} \leftrightarrow O$ ) and 405 cm<sup>–1</sup> is assigned as  $\nu_2$ . The band at 975 cm<sup>–1</sup> may be ascribed to the stretching vibration of Fe–Co. The absorption bands at 3,450 and 1,615 cm<sup>–1</sup> arise from the stretching and bending vibrations of hydroxyl groups, respectively. The band at 1,379 cm<sup>–1</sup> is ascribed to the anti-symmetric stretching vibration of NO<sub>3</sub><sup>–</sup>, arising from the residual nitrate.

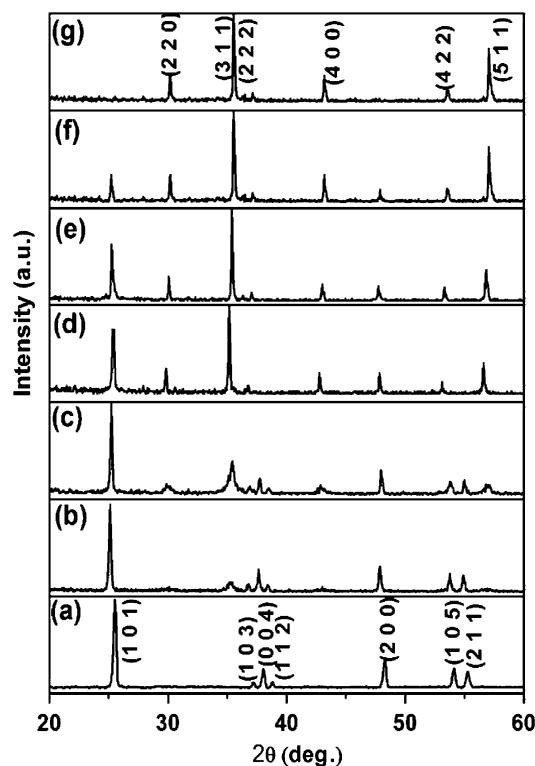
Figure 3c shows the FTIR spectra of as-synthesized  $x$ TiO<sub>2</sub> + (1 –  $x$ ) CoFe<sub>2</sub>O<sub>4</sub> (0.1 ≤  $x$  ≤ 0.9) composite powders. The band at 375–800 cm<sup>–1</sup> is attributed to the Ti–O–Ti or Fe–O stretching vibrations. It can be seen from the figure that as the mol% of TiO<sub>2</sub> is increasing, the intensity of the bands at 579 cm<sup>–1</sup> increases and shifts towards high frequency. The band became broad at high



**Fig. 3** FTIR spectra of microwave-hydrothermally synthesized **a**  $\text{TiO}_2$ , **b**  $\text{CoFe}_2\text{O}_4$  and **c**  $x\text{TiO}_2 + (1-x)\text{CoFe}_2\text{O}_4$  composites ( $0.1 \leq x \leq 0.9$ )

mol% of  $\text{TiO}_2$ . The band at  $405\text{ cm}^{-1}$  is disappearing when the mol% of  $\text{TiO}_2$  is increasing, indicates that  $\text{Ti}^{4+}$  ions occupy octahedral sites of spinel structure.

Figure 4 shows the XRD patterns of nanocomposite samples sintered at  $500^\circ\text{C}/30\text{ min}$ . It is clearly seen that two phases, ferrite and anatase, were identified in the composite samples. No intermediate phases such as  $\text{CoTiO}_3$  (JCPDS card no: 72-1069) and  $\text{Fe}_2\text{O}_3$  were observed in the X-ray analysis. This suggests that no significant chemical reactions were taken place during co-firing of the mixed powders, remaining the presence of distinct  $\text{TiO}_2$  and ferrite phases. This is very important for the preparation of ferrite +  $\text{TiO}_2$  composite materials, so that the dielectric properties of the composites could not degrade after sintering. It can also be observed that the number of ferrite peaks increases with an increase of ferrite content in



**Fig. 4** XRD patterns of microwave sintered  $x\text{TiO}_2 + (1-x)\text{CoFe}_2\text{O}_4$  composites ( $0 \leq x \leq 1$ ). **a**  $\text{TiO}_2$ , **b** CFT9, **c** CFT7, **d** CFT5, **e** CFT3, **f** CFT1 and **g** CF

the composite and vice versa. It clearly shows that the sintering at  $500^\circ\text{C}/30\text{ min}$  enhanced the intensity of composite diffraction peaks which indicates the improvement of crystallizability. The lattice parameters of the composites were given in Table 2. It can be seen from the table that the lattice parameters were increasing with an increase of  $\text{TiO}_2$  content.

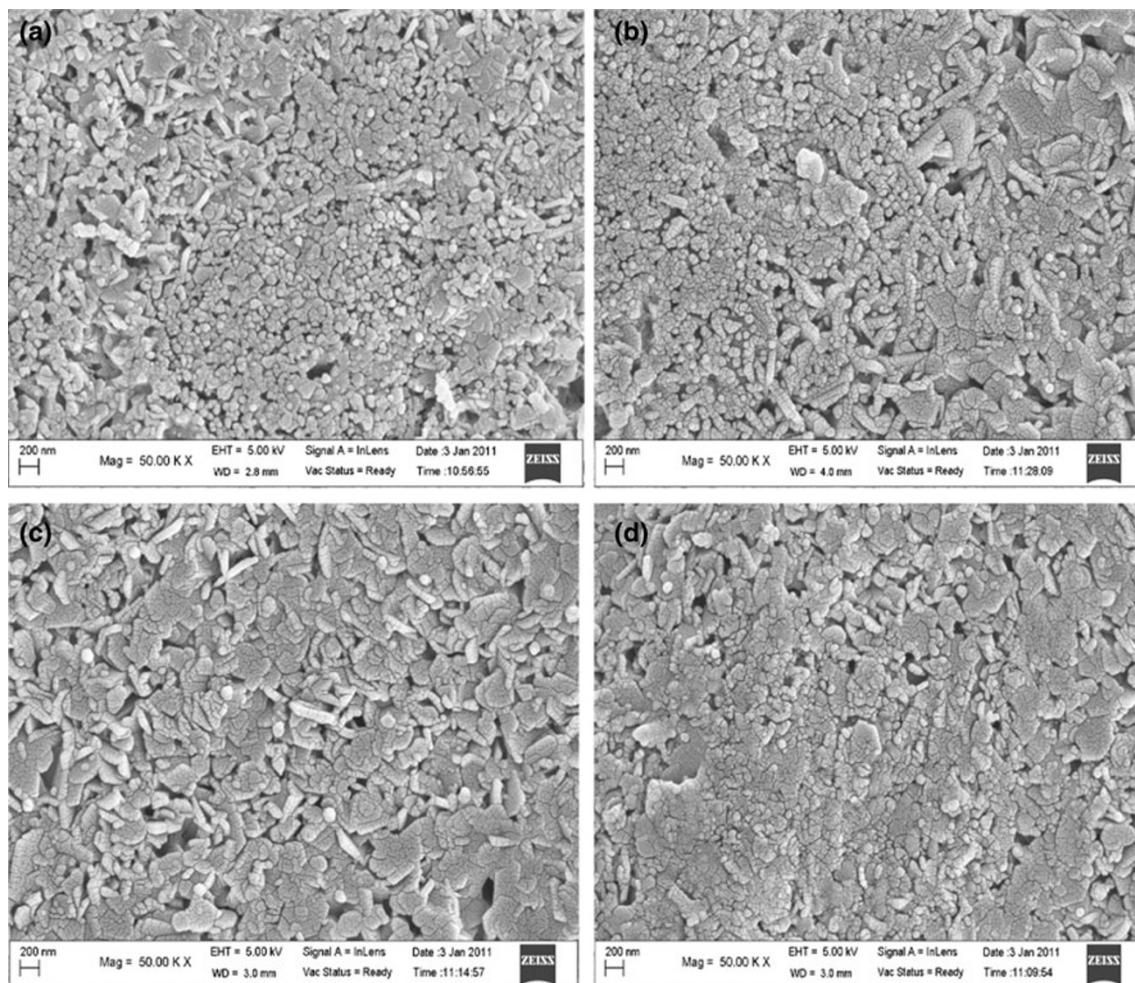
At  $500^\circ\text{C}$ ,  $\text{Ti}^{4+}$  ( $1.34\text{ \AA}$ ) substitute the  $\text{Fe}^{3+}$  ion ( $0.64\text{ \AA}$ ) at A and B sites of the spinel ferrite structure, according to the law of ion substitution.  $\text{Ti}^{4+}$  ions occupy the B sites of spinel ferrite structure; due to this, the lattice constant increases with an increase of  $\text{TiO}_2$  content. The lattice parameter of the  $\text{TiO}_2$  is not changed uniformly. The bulk density of the present sintered samples was measured using Archimedes's method and it was found to be increasing with an addition of  $\text{TiO}_2$ .

Figure 5a–d shows the FE-SEM images of the composites with different mol% of  $\text{TiO}_2$ . The microstructure properties like grain size, grain distribution and porosity significantly influence the properties of the composite materials. It is therefore essential to have a detailed microstructure analysis. In the images, the black grains are ferrite grains and the white ones are  $\text{TiO}_2$  grains. The values of grain sizes of ferrite were given in Table 2. It can be observed from the images that the grain size is in the range of  $54\text{--}78\text{ nm}$ . It can be seen that as



**Table 2** Data of lattice constant, bulk density,  $M_s$  and dielectric and magnetic properties of sintered samples

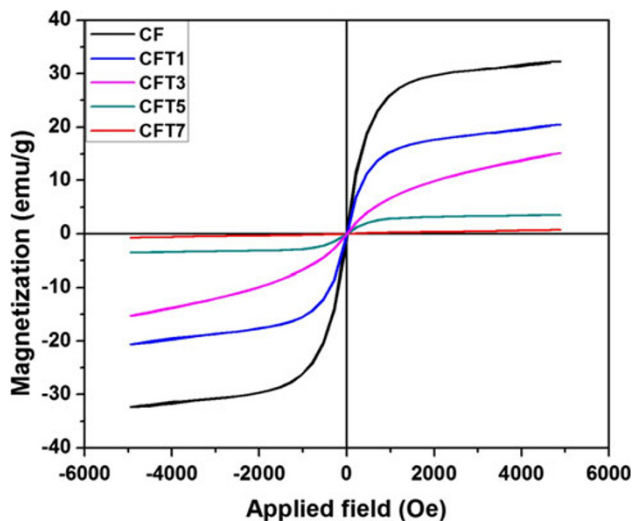
Sample name	Lattice constant			Bulk density (g/cm <sup>3</sup> )	Grain size of the ferrite (nm)	$M_s$ (emu/g)	At 1 MHz			
	Ferrite	TiO <sub>2</sub>					$\varepsilon'$	$\varepsilon''$	$\mu'$	$\mu''$
	(a) (Å)	(a) (Å)	(c) (Å)							
TO	–	3.773	9.509	–	–	–	–	–	–	–
CFT9	8.399	3.773	9.509	5.45	78	–	44	2.46	32	1.35
CFT7	8.397	3.773	9.511	5.42	72	–	36	2.21	71	1.67
CFT5	8.395	3.774	9.512	5.39	66	4	30	2.16	110	1.98
CFT3	8.394	3.774	9.512	5.35	60	10	24	1.85	172	2.27
CFT1	8.394	3.774	9.514	5.33	54	22	19	1.46	398	2.52
CF	8.393	–	–	5.30	–	34	14	1.12	773	2.84

**Fig. 5** SEM images of microwave sintered  $x\text{TiO}_2 + (1 - x) \text{CoFe}_2\text{O}_4$  composites ( $0 \leq x \leq 1$ ). **a** CFT3, **b** CFT5, **c** CFT7 and **d** CFT9

the  $\text{TiO}_2$  content increasing, the samples are becoming dense. The sample CFT9 shows less pores and the distribution of the grains is uniform.

Figure 6 shows the magnetic hysteresis loops of  $x\text{TiO}_2 + (1 - x) \text{CoFe}_2\text{O}_4$  nanocomposites with  $x = 0$ ,

0.1, 0.3, 0.5 and 0.7 mol%, respectively, indicating that the composites are magnetically ordered. The values of saturation magnetization ( $M_s$ ) were given in Table 2. It is observed that the values of saturation magnetization of the composites were found to be decreasing with an increase of

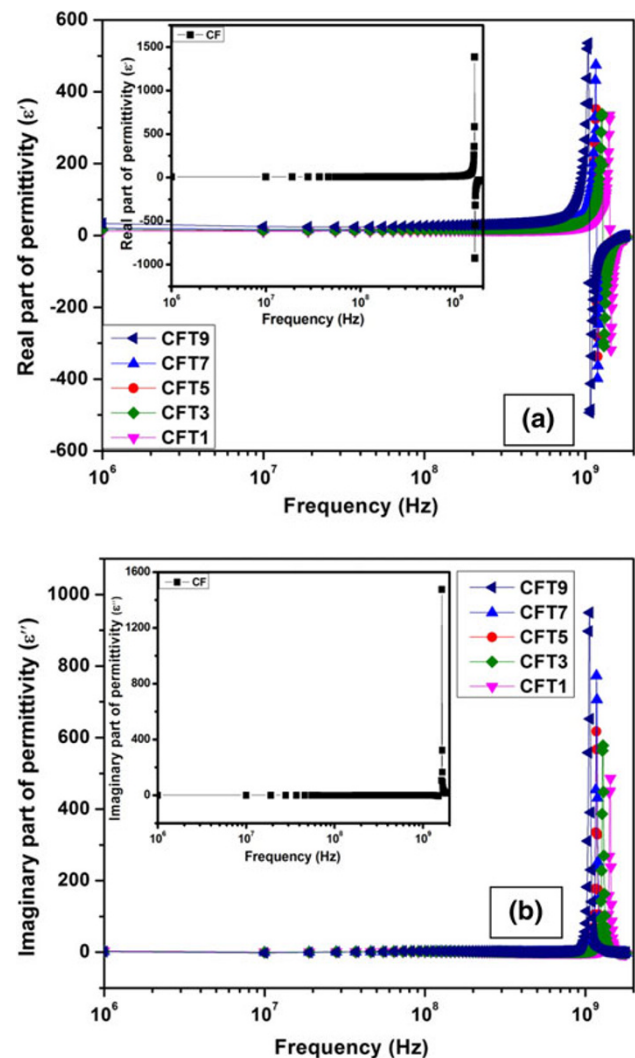


**Fig. 6** VSM loops of  $\text{TiO}_2 + \text{CoFe}_2\text{O}_4$  nanocomposites

$\text{TiO}_2$  due to the dilution effect of non-magnetic  $\text{TiO}_2$ . The saturation magnetization of  $\text{TiO}_2$  is unity due to its inherent non-magnetic nature.

The frequency variation of real ( $\epsilon'$ ) and imaginary ( $\epsilon''$ ) parts of permittivity for all the samples under investigation was measured in the frequency range of 1 MHz–1.8 GHz and obtained results are plotted in Fig. 7a and b. It can be seen from the figures that the value of  $\epsilon'$  remains constant up to 300 MHz and increases further increase of frequency. In all the samples, a resonance peak was observed around 1 GHz. This behavior can be explained in the following way: the  $\epsilon'$  remains constant in the frequency range from 1 to 300 MHz due to the hopping electrons will not follow the external applied field. Whereas, the increase of  $\epsilon'$  around 1 GHz is may be due to the following of hopping electrons with the external field. When the hopping frequency of the electrons is equal to that of the external applied electric field, a peak obtained is called as the dielectric resonance.

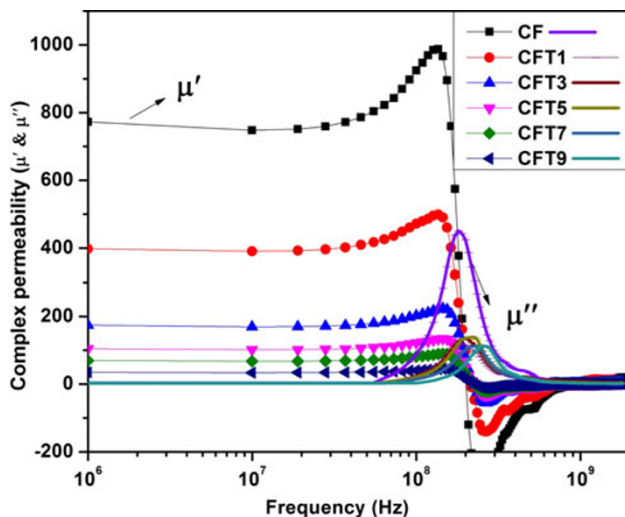
As  $\text{TiO}_2$  increases from  $x = 0.1$  to 0.9, the values of  $\epsilon'$  also increases from 19 to 44 at 1 MHz and the values were given in Table 2. The increase of real part of permittivity ( $\epsilon'$ ) is attributed to the introduction of  $\text{TiO}_2$  which raises the amount of dipoles in the composite materials. The increase of dipoles tends to increase the local displacements (dielectric polarization) in the direction of external applied electric field for electrons, and the increased polarization causes a significant enhancement of the dielectric constant. The frequency dependence of imaginary part of permittivity ( $\epsilon''$ ) has been measured on all the composites in the range of 1 MHz–1.8 GHz and obtained results were plotted in Fig. 7b. It can be seen from the figure that the value of  $\epsilon'$  is small and remains almost constant from 1 to 300 MHz. The  $\epsilon''$  value increases with an increase of  $\text{TiO}_2$  content. The variation of  $\epsilon''$  with frequency may be explained



**Fig. 7** Frequency dependence of **a** real ( $\epsilon'$ ) and **b** imaginary ( $\epsilon''$ ) parts of permittivity

similar to that of  $\epsilon'$  variation with frequency. The change in the  $\epsilon'$  and  $\epsilon''$  with frequency for  $\text{TiO}_2\text{--CoFe}_2\text{O}_4$  composites obeys Lichtenecker's mixed law (Kondo et al. 1999).

Figure 8 shows the frequency dependence of complex permeability for composite samples at room temperature. It was observed from the figures that the real ( $\mu'$ ) part of permeability remained almost constant, until the frequency was raised to a certain value and then began to decrease at higher frequency. The imaginary ( $\mu''$ ) part of permeability gradually increased with the frequency and took a maximum at a certain frequency, where the  $\mu'$  rapidly decreases. This feature is well known as natural resonance. As the wt% of  $\text{TiO}_2$  increases from  $x = 0.1$  to 0.9, the real part of permeability ( $\mu'$ ) decreases to very small value at 1 MHz, and the values were given in Table 2. It can be observed from the figure that the magnetic resonance frequency for the composite increases with decrease in ferrite content.



**Fig. 8** Frequency dependence of real ( $\mu'$ ) and imaginary ( $\mu''$ ) parts of permeability

This may be attributed to the decrease in the interaction between ferrite particles due to the dilute effect of non-magnetic particles. With an increase of  $\text{TiO}_2$ , the natural resonance frequency having a maximum value shifted towards high frequency.

The  $\mu''$  of  $\text{TiO}_2 + \text{CoFe}_2\text{O}_4$  composites are higher than that of pure  $\text{CoFe}_2\text{O}_4$  in the whole frequency range. The result can be explained by magnetic dissipation. According to Van der Zaag's suggestion (Van der Zaag 1999), the magnetic dissipation of ferrites can be classified as: hysteresis loss, eddy current loss, residual loss, ferromagnetic resonance loss and intragranular domain wall loss. In this study, however, the enhancement of eddy loss might be contributed to higher  $\mu''$  of  $\text{TiO}_2 + \text{CoFe}_2\text{O}_4$  nanocomposites, because of  $\text{TiO}_2$  being a semiconductor with band gap energy of 3.2 eV (Zuo and Victoria 2003; Lee et al. 2004). Especially, when the weight ratio of  $\text{TiO}_2$  is 20 %, both  $\mu'$  and  $\mu''$  of  $\text{TiO}_2 + \text{CoFe}_2\text{O}_4$  nanocomposites show maximum value.

## Conclusions

For the first time  $x\text{TiO}_2 + (1-x)\text{CoFe}_2\text{O}_4$  nanocomposites were synthesized using microwave-hydrothermal method. The XRD shows the single phase formation of spinel and  $\text{TiO}_2$  phases, respectively. High dense, homogeneous and small grained  $\text{CoFe}_2\text{O}_4 + \text{TiO}_2$  were prepared using microwave sintering method at 500 °C/30 min. The variation of lattice constant was explained using structural formula. The average grain size of all the composites lies between 54 and 78 nm. The real permittivity and permeability values were not much affected by the addition of  $\text{TiO}_2$  content, but the resonant frequency of all the sintered samples found to be >1 GHz.

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